PATENT ABSTRACTS OF JAPAN

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(54) METHOD AND APPARATUS FOR COATING CERAMIC MONOLITHIC SUPPORT WITH CATALYTIC SLURRY

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a method and an apparatus for coating a ceramic monolithic support with a catalytic slurry wherein a deviation in the upper and lower sides of a coating amount can be minimized in coating the monolithic support.
SOLUTION: In the case where a monolithic support which is not coated by anything is placed and pressurized or sucked, conditions for pressure and time are previously set so that pressure is stepwise or continuously changed and so that all pressurization or suction time is made T hours, an x1 pressure is applied for initial t1 hours, an x2 pressure is generated for the next t2 hours, and an x3 pressure is generated for the succeeding t3 hours, wherein, $T=t1+t2+t3+...+tn(n\geq 2)$.

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* NOTICES *

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CLAIMS

[Claim(s)]
[Claim 1] How to coat with a catalyst slurry the ceramic monolith support characterized by changing two or more steps of welding pressure, and coating it in the pressurization time amount T in the approach of being the approach of coating the internal surface of ceramic monolith support with a catalyst slurry, making face the one direction of a tubular path a catalyst slurry, and coating by pressurization pushing from an one direction.
[Claim 2] How to coat with a catalyst slurry the ceramic monolith support characterized by changing two or more steps of suction pressure, and coating it in the reduced pressure suction time amount T in the approach of being the approach of coating the internal surface of ceramic monolith support with a catalyst slurry, making face the one direction of a tubular path a catalyst slurry, and coating by reduced pressure suction from the other side.
[Claim 3] How to coat with a catalyst slurry the ceramic monolith support characterized by to include the damper of the stepping motor drive which operates as the opening beforehand set up in pressurization piping, to control the opening of the damper of said stepping motor drive, to change two or more steps of said welding pressure, and to coat it in the pressurization time amount T as a means change said welding pressure in the approach of coating ceramic monolith support according to claim 1 with a catalyst slurry.
[Claim 4] How to coat with a catalyst slurry the ceramic monolith support characterized by making it open to the time amount which prepared one or more closing motion bulbs in the pressurization piping side face, and was set up beforehand, changing two or more steps of said welding pressure, and coating it in the pressurization time amount T as a means to change said welding pressure, in the approach of coating ceramic monolith support according to claim 1 with a catalyst slurry.
[Claim 5] How to coat with a catalyst slurry the ceramic monolith support characterized by to include the damper of the stepping motor drive which operates as the opening beforehand set up in suction piping, to control the opening of the damper of said stepping motor drive, to change two or more steps of suction pressure, and to coat it in the reduced-pressure suction time amount T as a means change said suction pressure in the approach of coating ceramic monolith support according to claim 2 with a catalyst slurry.
[Claim 6] How to coat with a catalyst slurry the ceramic monolith support characterized by making it open to the time amount which prepared one or more closing motion bulbs in suction piping or a suction tub, and was set up beforehand, changing two or more steps of suction pressure, and coating it in the reduced pressure suction time amount T as a means to change said suction pressure, in the approach of coating ceramic monolith support according to claim 2 with a catalyst slurry.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the approach of forming a catalyst bed in a detail at homogeneity at ceramic monolith support, and its equipment further about the approach of forming a catalyst bed in ceramic monolith support, and its equipment.
[0002]
[Description of the Prior Art] As the catalyst bed formation approach of the conventional ceramic monolith support As indicated by JP,62-28695,B for example, on the tubular path of ceramic monolith support What forms a sink and a catalyst bed for a catalyst slurry using vaccum pressure, and ceramic monolith support are immersed in a catalyst slurry. There are some which form a sink and a catalyst bed about the inside of a tubular path by positioning a catalyst slurry to what forms a catalyst bed, and the upper limit of ceramic monolith support, and applying a pressure to this catalyst slurry.
[0003]
[Problem(s) to be Solved by the Invention] Recently, for exhaust gas purification, a catalyst bed is thickened, moreover it raises the engine performance, it is made multilayer structure, and it thickens, and the approach of raising the engine performance is taken increasingly.
[0004] However, in some which use vaccum pressure for a catalyst slurry and form a sink and a catalyst bed on the aforementioned conventional approach, for example, the tubular path of ceramic monolith support, there is a problem that the thickness of a catalyst bed differs, an entry side is thin and an exhaust port side becomes thick, by the exhaust port side of the entry side of the catalyst slurry of ceramic monolith support, and a catalyst slurry.
[0005] Even if it is in some which similarly position a catalyst slurry to the upper limit of ceramic monolith support, apply a pressure to this catalyst slurry, and form a sink and a catalyst bed for the inside of a tubular path, there is a problem that the thickness of a catalyst bed differs, an entry side is thin and an exhaust port side becomes thick, by the exhaust port side of the entry side of a catalyst slurry, and a catalyst slurry.
[0006] The ununiformity of this catalyst bed thickness bars stabilization of the catalyst engine performance, and the appearance of the approach of forming a catalyst bed in uniform thickness has faced it.
[0007] The purpose of this invention solves the above-mentioned technical problem, and is to offer the approach of forming a catalyst bed in uniform thickness.
[0008]
[Means for Solving the Problem and its Function] In applying a catalyst slurry to ceramic monolith support, and forming a catalyst bed, this invention controls welding pressure and suction pressure, and relates to the coating approach and coating equipment which equalize the thickness (amount) of a catalyst bed.
[0009] this invention persons found out the coating approach which hardly changes the thickness (amount) of a catalyst bed by the exhaust port side of a catalyst slurry the entry side of a catalyst slurry by pressurization or control of suction pressure as a result of the research on the coating approach of the catalyst slurry to ceramic monolith support. Moreover, the equipment which makes this coating possible was resulted in a header and this invention.
[0010] The catalyst slurry was impressed to one side of ceramic monolith support, and when having pressurized or drawn in and having been seen on the conditions which place conventionally the monolith support which nothing has coated, and are pressurized or attracted, the approach in which a fixed pressure carries out a fixed time amount load was taken. In order to lengthen by the fixed pressure in this approach even after a catalyst slurry falls the path of ceramic monolith support out, a path is narrow by the slurry (catalyst bed) by which coating was carried out, and ventilation resistance becomes large, and the pressure

of this air also rises compared with the case where the monolith support which nothing has coated is placed, to a catalyst bed, and turns into force which depresses a catalyst bed downward to it. For this reason, by the entry side of a catalyst slurry, a catalyst bed is thin and becomes thick by the exhaust port side of a catalyst slurry. (Refer to drawing 6 (a))

[0011] In this invention, immediately after a catalyst slurry falls the path of ceramic monolith support out, or it reduces pressurization or suction pressure gradually, it is made to fall continuously and pressurization and suction are terminated. According to this, the pressure concerning a catalyst bed will be lowered, the way of the viscosity of a catalyst bed becomes it is strong and possible [stopping at the location], and a uniform catalyst bed can be obtained. (Refer to drawing 6 (b))

[0012]

[Embodiment of the Invention] Hereafter, the gestalt of operation of the approach of coating the ceramic monolith support by this invention with a catalyst slurry and its equipment is explained to a detail. It is the approach of coating the internal surface of ceramic monolith support with a catalyst slurry, and this invention makes a catalyst slurry face the one direction of a tubular path, it carries out pressurization pushing from an one direction, or relates to the technique which carries out reduced pressure suction from the other side. When specifically placing the monolith support which nothing has coated and pressurizing or drawing in, As whole pressurization or suction time amount is made into T hours, the load of the pressure of x1 is carried out for t 1 hour of the start, the pressure of x2 is produced for the following t 2 hours and the pressure of x3 is produced for the following t 3 hours A pressure and time amount conditions are beforehand set up so that a pressure may be changed gradually or continuously. Here, they are T=t1+t2+t3+ and +tn (n>=2).

[0013] Hereafter, an example and the example of a comparison explain concretely the gestalt of operation of the approach of coating the ceramic monolith support by this invention with a catalyst slurry, and its equipment.

[0014] (Example 1 of a comparison) 82.8g [of things which supported Rh 2% to gamma alumina], 542.9g [of things which supported Pd 3% to gamma alumina], 72.7g [of gamma alumina], and boehmite alumina 29.6g, 216g of 10% nitric acids, and 1072g of water were put into the ball mill, it ground for 90 minutes, and the mean particle diameter of 3.5micro and the catalyst slurry of viscosity 70cP were prepared at 40% of solid content. Water was added to this and it considered as 33% of solid content, and the catalyst slurry of viscosity 33cP.

[0015] The monolith support (capacity 1.7L: 2x15cm with an ellipse of 113cm) which nothing has coated was put on the suction tub, and was attracted, and suction conditions were set up so that the pressure of -260mmAq might be obtained. The 800g of the above-mentioned slurries was impressed to monolith support (capacity 1.7L: 2x15cm with an ellipse of 113cm), it drew in for 10 seconds by the flow and pressure requirement of -260mmAq, and the monolith support to which the catalyst bed was attached was calcinated at 400 degrees C after desiccation by 120 degrees C for 1 hour. Thereby, the alumina system catalyst became the monolith support by which 100 g/L coating was carried out.

[0016] When this monolith support with a catalyst was equally divided into three under Kaminaka and these amounts of coatings were investigated, in the upper part, they were 115 g/L in 100g/L, and the lower part in 85g/L, and pars intermedia.

[0017] (Example 2 of a comparison) 540g of water and silica sol (SiO2 : 20% content) 450g were added to zeolite 810g, the ball mill ground for 120 minutes, and the zeolitic-catalyst slurry was prepared. The solid content of the slurry at this time was 50%, and was the mean particle diameter of 4.2micro, and viscosity 32cP.

[0018] The monolith support (capacity 1.3L: 2x11.5cm with an ellipse of 113cm) which nothing has coated was put on the suction tub, and was attracted, and suction conditions were set up so that the pressure of -280mmAq might be obtained. The 700g of the above-mentioned slurries was impressed to monolith support (capacity 1.3L: 2x11.5cm with an ellipse of 113cm), it drew in for 10 seconds by the flow and pressure requirement of -280mmAq, and the monolith support to which the catalyst bed was attached was calcinated at 400 degrees C after desiccation by 120 degrees C for 1 hour. Thereby, the zeolitic catalyst became the monolith support by which 200 g/L coating was carried out.

[0019] When this monolith support with a catalyst was equally divided into three under Kaminaka and these amounts of coatings were investigated, in the upper part, they were 225 g/L in 205g/L, and the lower part in 170g/L, and pars intermedia.

[0020] (Example 3 of a comparison) 421.2g [of things which sank Pd into gamma alumina 3%], 288g [of gamma alumina], and boehmite alumina 14.8g, and 1076g of water were put into the ball mill, it ground for

150 minutes, and the mean particle diameter of 3.0micro and the catalyst slurry of viscosity 110cP were prepared at 40% of solid content. Water was added to this and it considered as 35% of solid content, and the catalyst slurry of viscosity 56cP.
[0021] The monolith support (capacity 1.7L: 2x15cm with an ellipse of 113cm) which nothing has coated was put on the suction tub, and was attracted, and suction conditions were set up so that the pressure of -260mmAq might be obtained. The 700g of the above-mentioned slurries was impressed to monolith support (capacity 1.7L: 2x15cm with an ellipse of 113cm), it drew in for 10 seconds by the flow and pressure requirement of -260mmAq, and the monolith support to which the catalyst bed was attached was calcinated at 400 degrees C after desiccation by 120 degrees C for 1 hour. Thereby, the alumina system catalyst became the monolith support by which 100 g/L coating was carried out.
[0022] When this monolith support with a catalyst was equally divided into three under Kaminaka and these amounts of coatings were investigated, in the upper part, they were 110 g/L in 105g/L, and the lower part in 85g/L, and pars intermedia.
[0023] (Example 1) 82.8g [of things which supported Rh 2% to gamma alumina], 542.9g [of things which supported Pd 3% to gamma alumina], 72.7g [of gamma alumina], and boehmite alumina 29.6g, 216g of 10% nitric acids, and 1072g of water were put into the ball mill, it ground for 90 minutes, and the mean particle diameter of 3.5micro and the catalyst slurry of viscosity 70cP were prepared at 40% of solid content. Water was added to this and it considered as the same catalyst slurry as 33% of solid content, and the example 1 of a comparison of viscosity 33cP.
[0024] The monolith support (capacity 1.7L: 2x15cm with an ellipse of 113cm) which nothing has coated was put on the suction tub, and suction conditions as shown in drawing 1 were set up. That is, for [of the start] 2 seconds, it set up so that it might draw in by -180mmAq for [of -260mmAq and a degree] 4 seconds and might draw in by -100mmAq for [of the last] 4 seconds. The 800g of the above-mentioned slurries was impressed to monolith support (capacity 1.7L: 2x15cm with an ellipse of 113cm), and coating was performed on the suction conditions of drawing 1 . The monolith support to which this catalyst bed was attached was calcinated at 400 degrees C after desiccation by 120 degrees C for 1 hour. Thereby, the alumina system catalyst became the monolith support by which 100 g/L coating was carried out.
[0025] When this monolith support with a catalyst was equally divided into three under Kaminaka and these amounts of coatings were investigated, in the upper part, they were 102 g/L in 100g/L, and the lower part in 98g/L, and pars intermedia.
[0026] (Example 2) 540g of water and silica sol (SiO_2 : 20% content) 450g were added to zeolite 810g, the ball mill ground for 120 minutes, and the zeolitic-catalyst slurry was prepared. The solid content of the slurry at this time is 50%, is the mean particle diameter of 4.2micro, and viscosity 32cP, and is the same slurry as the example 2 of a comparison.
[0027] The monolith support (capacity 1.3L: 2x11.5cm with an ellipse of 113cm) which nothing has coated was put on the suction tub, and suction conditions as shown in drawing 2 were set up. That is, for [of the start] 2 seconds, it set up so that it might draw in by -180mmAq for [of -280mmAq and a degree] 4 seconds and might draw in by -100mmAq for [of the last] 4 seconds. The 700g of the above-mentioned slurries was impressed to monolith support (capacity 1.3L: 2x11.5cm with an ellipse of 113cm), and coating was performed on the suction conditions of drawing 2 . The monolith support to which this catalyst bed was attached was calcinated at 400 degrees C after desiccation by 120 degrees C for 1 hour. Thereby, the zeolitic catalyst became the monolith support by which 200 g/L coating was carried out.
[0028] When this monolith support with a catalyst was equally divided into three under Kaminaka and these amounts of coatings were investigated, in the upper part, they were 203 g/L in 201g/L, and the lower part in 196g/L, and pars intermedia.
[0029] (Example 3) 421.2g [of things which sank Pd into gamma alumina 3%], 288g [of gamma alumina], and boehmite alumina 14.8g, and 1076g of water were put into the ball mill, it ground for 150 minutes, and the mean particle diameter of 3.0micro and the catalyst slurry of viscosity 110cP were prepared at 40% of solid content. Water was added to this and it considered as the same catalyst slurry as 35% of solid content, and the example 3 of a comparison of viscosity 56cP.
[0030] The monolith support (capacity 1.7L: 2x15cm with an ellipse of 113cm) which nothing has coated was put on the suction tub, and suction conditions as shown in drawing 3 were set up. That is, for [of the start] 2 seconds, it set up so that it might draw in by -180mmAq for [of -260mmAq and a degree] 4 seconds and might draw in by -100mmAq for [of the last] 4 seconds. The 700g of the above-mentioned slurries was impressed to monolith support (capacity 1.7L: 2x15cm with an ellipse of 113cm), and coating was performed on the suction conditions of drawing 3 . The monolith support to which this catalyst bed was

attached was calcinated at 400 degrees C after desiccation by 120 degrees C for 1 hour. Thereby, the alumina system catalyst became the monolith support by which 100 g/L coating was carried out.
[0031] When this monolith support with a catalyst was equally divided into three under Kaminaka and these amounts of coatings were investigated, in the upper part, they were 101.5 g/L in 100g/L, and the lower part in 98.5g/L, and pars intermedia.
[0032] (Example 4) As an example of the system which has materialized this invention, the equipment which performs coating by reduced pressure suction is shown in drawing 4 . The process shown here is a semi-automatic process, and an operator puts in by hand the monolith support by which coating should be carried out first in a system, and after coating of it is carried out, he has to take out.
[0033] A general view of a process step performed by this system is as follows. An operator puts the monolith support by which coating should be carried out on the coating tub 11, and turns on a main switch (breaker) 1, the blower start switch 2, and the operation preparation switch 4. Next, the time amount holding the opening and opening of the suction pressure control damper (damper opening is controlled by the stepping motor) 13 is inputted on the actuation screen 9 on a control panel 12. Then, suction will be stopped if it becomes the time amount which suction was started and was set up when the unattended operation initiation switch 5 was turned on.
[0034] Although the suction pressure and suction time amount at this time were expressed, examples are above-mentioned drawing 1 - drawing 3 . This condition does not disappear until it is remembered to input once and inputs another conditions. For this reason, coating in the same conditions can be repeated and can be performed.
[0035] Next, an operator attaches a hopper in the upper part of monolith support, into this, supplies the catalyst slurry prepared beforehand and turns on the unattended operation initiation switch 5 after this. If suction like the example shown in drawing 1 - drawing 3 is performed and it becomes the setup time, suction will be stopped and coating will be completed. An operator removes a hopper from the monolith support which ended coating, and takes out monolith support, and an activity ends him.
[0036] (Example 5) As another example of the system which has materialized this invention, the equipment which performs coating by reduced pressure suction is shown in drawing 5 . The process shown here is a semi-automatic process, and an operator puts in by hand the monolith support by which coating should be carried out first in a system, and after coating of it is carried out, he has to take out.
[0037] A general view of a process step performed by this system is as follows. An operator puts the monolith support by which coating should be carried out on the coating tub 11, and turns on a main switch (breaker) 1, the blower start switch 2, and the operation preparation switch 4. Next, the time amount which opens the suction pressure control bulbs 16, 17, and 18 is inputted on the actuation screen 9 on a control panel 12. Suction will be stopped if it becomes the time amount which suction was started and was set up when total suction time amount was inputted and the unattended operation initiation switch 5 was turned on after this. Since the open air is attracted by the coating tub 11 by opening the suction pressure control bulbs 16, 17, and 18, the pressure in the coating tub 11 declines. Not only one kind but the thing equipped with two or more kinds of the magnitude (path) and the number of this bulb is desirable. This is because suction force differs with the path of a bulb, and the direction with much number of a bulb is because the conditions of suction can be changed finely.
[0038] Although the suction pressure and suction time amount at this time were expressed, examples are above-mentioned drawing 1 - drawing 3 . This condition does not disappear until it is remembered to input once and inputs another conditions. For this reason, coating in the same conditions can be repeated and can be performed.
[0039] Next, an operator attaches a hopper in the upper part of monolith support, into this, supplies the catalyst slurry prepared beforehand and turns on the unattended operation initiation switch 5 after this. If suction like the example shown in drawing 1 - drawing 3 is performed and it becomes the setup time, suction will be stopped and coating will be completed. An operator removes a hopper from the monolith support which ended coating, and takes out monolith support, and an activity ends him.
[0040]
[Effect of the Invention] As mentioned above, as explained to the detail, according to this invention, in coating to monolith support, the effectiveness that the bias in the upper and lower sides of the amount of coatings can be made very small is acquired. That is, the catalyst of uniform thickness can be acquired.

[Translation done.]

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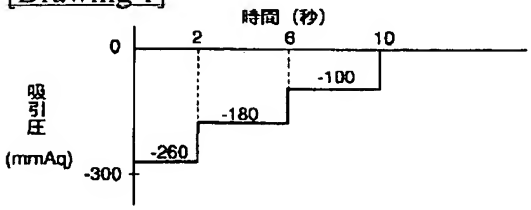
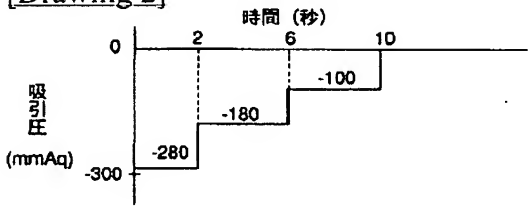
DRAWINGS

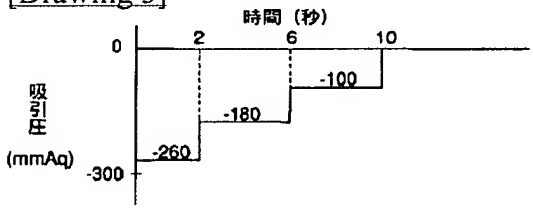
[Drawing 1]



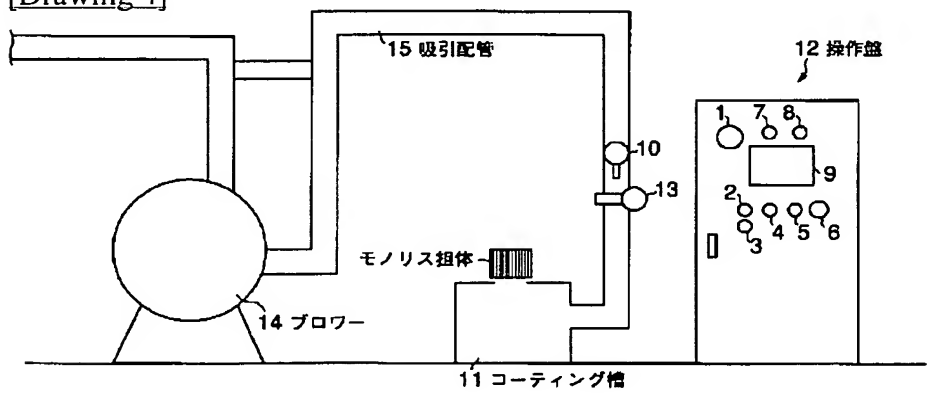
[Drawing 2]



[Drawing 3]



[Drawing 4]



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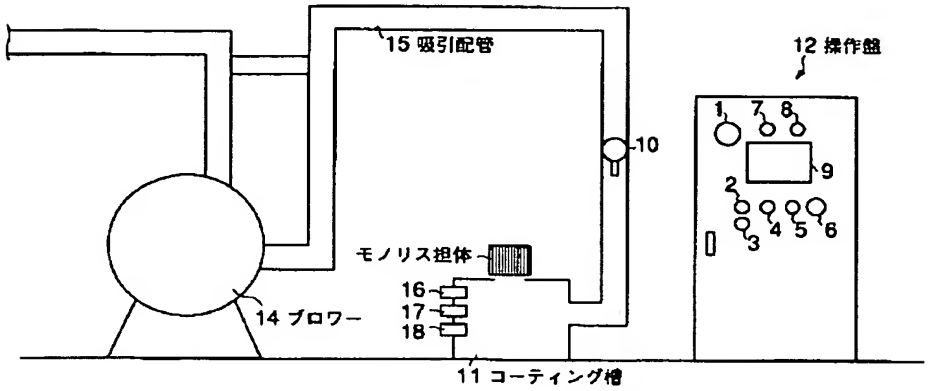
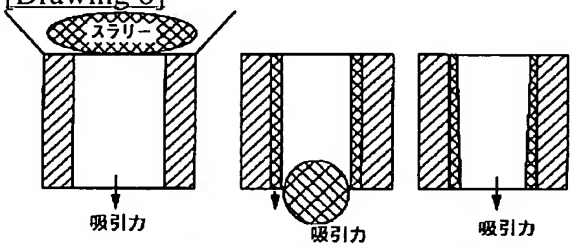
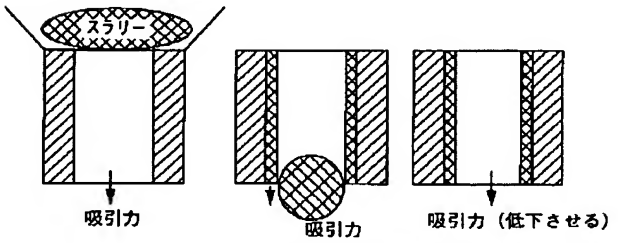


[Drawing 6]



(a) 従来技術による触媒層の形成（減圧吸引の場合）



(b) 本発明による触媒層の形成（減圧吸引の場合）

[Translation done.]